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NEWS	1		Web Page for STN Seminar Schedule - N. America
NEWS	2	DEC 01	ChemPort single article sales feature unavailable
NEWS	3	JAN 06	The retention policy for unread STNmail messages will change in 2009 for STN-Columbus and STN-Tokyo
NEWS	4	JAN 07	WPIDS, WPINDEX, and WPIX enhanced Japanese Patent Classification Data
NEWS	5	FEB 02	Simultaneous left and right truncation (SLART) added for CERAB, COMPUAB, ELCOM, and SOLIDSTATE
NEWS	6	FEB 02	GENBANK enhanced with SET PLURALS and SET SPELLING
NEWS	7	FEB 06	Patent sequence location (PSL) data added to USGENE
NEWS	8	FEB 10	COMPENDEX reloaded and enhanced
NEWS	9	FEB 11	WTEXTILES reloaded and enhanced
NEWS	10	FEB 19	New patent-examiner citations in 300,000 CA/Caplus patent records provide insights into related prior art
NEWS	11	FEB 19	Increase the precision of your patent queries -- use terms from the IPC Thesaurus, Version 2009.01
NEWS	12	FEB 23	Several formats for image display and print options discontinued in USPATFULL and USPAT2
NEWS	13	FEB 23	MEDLINE now offers more precise author group fields and 2009 MeSH terms
NEWS	14	FEB 23	TOXCENTER updates mirror those of MEDLINE - more precise author group fields and 2009 MeSH terms
NEWS	15	FEB 23	Three million new patent records blast AEROSPACE into STN patent clusters
NEWS	16	FEB 25	USGENE enhanced with patent family and legal status display data from INPADOCDB
NEWS	17	MAR 06	INPADOCDB and INPAFAMDB enhanced with new display formats
NEWS	18	MAR 11	EPFULL backfile enhanced with additional full-text applications and grants
NEWS	19	MAR 11	ESBIOBASE reloaded and enhanced
NEWS	20	MAR 20	CAS databases on STN enhanced with new super role for nanomaterial substances
NEWS	21	MAR 23	CA/Caplus enhanced with more than 250,000 patent equivalents from China
NEWS	22	MAR 30	IMSPATENTS reloaded and enhanced
NEWS	23	APR 03	CAS coverage of exemplified prophetic substances enhanced
NEWS	24	APR 07	STN is raising the limits on saved answers

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NEWS EXPRESS JUNE 27 08 CURRENT WINDOWS VERSION IS V8.3,  
AND CURRENT DISCOVER FILE IS DATED 23 JUNE 2008.

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NEWS IPC8 For general information regarding STN implementation of IPC 8

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\* \* \* \* \* STN Columbus \* \* \* \* \*

FILE 'HOME' ENTERED AT 16:56:55 ON 21 APR 2009

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FULL ESTIMATED COST	0.22	0.22

FILE 'REGISTRY' ENTERED AT 16:57:07 ON 21 APR 2009

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STRUCTURE FILE UPDATES: 20 APR 2009 HIGHEST RN 1137276-53-9  
DICTIONARY FILE UPDATES: 20 APR 2009 HIGHEST RN 1137276-53-9

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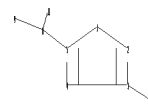
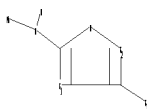
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predicted properties as well as tags indicating availability of experimental property data in the original document. For information on property searching in REGISTRY, refer to:

<http://www.cas.org/support/stngen/stndoc/properties.html>

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chain nodes :  
8 9 11 13  
ring nodes :  
1 2 3 4 5  
chain bonds :  
3-11 5-8 8-9 8-13  
ring bonds :  
1-2 1-5 2-3 3-4 4-5  
exact/norm bonds :  
1-2 1-5 2-3 3-4 3-11 4-5 5-8 8-9 8-13  
isolated ring systems :  
containing 1 :

G1:S,CH

G2:C,N

G3:Ph,Cy,Hy

Match level :  
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 8:CLASS 9:CLASS 11:CLASS 13:CLASS

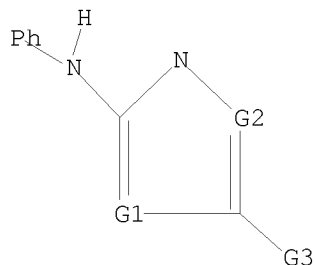
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L1 STRUCTURE UPLOADED

=> d l1

L1 HAS NO ANSWERS

L1 STR



G1 S,CH

G2 C,N

G3 Ph,Cy,Hy

Structure attributes must be viewed using STN Express query preparation.

=> s l1

SAMPLE SEARCH INITIATED 16:57:24 FILE 'REGISTRY'

SAMPLE SCREEN SEARCH COMPLETED - 26838 TO ITERATE

7.5% PROCESSED 2000 ITERATIONS 0 ANSWERS  
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)  
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
BATCH \*\*COMPLETE\*\*  
PROJECTED ITERATIONS: 526956 TO 546564  
PROJECTED ANSWERS: 0 TO 0

L2 0 SEA SSS SAM L1

=> s l1 sss full

FULL SEARCH INITIATED 16:57:31 FILE 'REGISTRY'

FULL SCREEN SEARCH COMPLETED - 540616 TO ITERATE

100.0% PROCESSED 540616 ITERATIONS 16 ANSWERS  
SEARCH TIME: 00.00.08

L3 16 SEA SSS FUL L1

=> FIL HCAPLUS

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

185.88

186.10

FILE 'HCAPLUS' ENTERED AT 16:57:46 ON 21 APR 2009

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FILE COVERS 1907 - 21 Apr 2009 VOL 150 ISS 17  
FILE LAST UPDATED: 20 Apr 2009 (20090420/ED)

HCAplus now includes complete International Patent Classification (IPC) reclassification data for the third quarter of 2008.

CAS Information Use Policies apply and are available at:

<http://www.cas.org/legal/infopolicy.html>

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 13

L4 14 L3

=> s 14 and py<=2003

24035193 PY<=2003

L5 9 L4 AND PY<=2003

=> d 14 ibib abs hitstr tot

L4 ANSWER 1 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2007:1059573 HCAPLUS

DOCUMENT NUMBER: 147:469265

TITLE: Structure-Based Design and Synthesis of  
(5-Arylamino-2H-pyrazol-3-yl)-biphenyl-2',4'-diols as  
Novel and Potent Human CHK1 Inhibitors

AUTHOR(S): Teng, Min; Zhu, Jinjiang; Johnson, Michael D.; Chen,  
Ping; Kornmann, Jill; Chen, Enhong; Blasina,  
Alessandra; Register, James; Anderes, Kenna; Rogers,  
Caroline; Deng, Yali; Ninkovic, Sacha; Grant, Stephan;  
Hu, Qiyue; Lundgren, Karen; Peng, Zhengwei; Kania,  
Robert S.

CORPORATE SOURCE: Department of Medicinal Chemistry, Biochemical  
Pharmacology, Research Pharmacology, Crystallography  
and Computational Chemistry, Pfizer Global Research  
and Development, San Diego, CA, 92121-1194, USA

SOURCE: Journal of Medicinal Chemistry (2007), 50(22),  
5253-5256

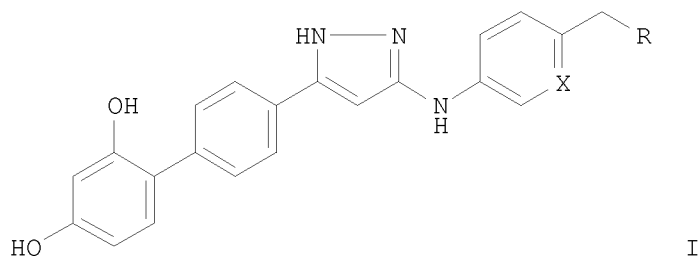
CODEN: JMCMAR; ISSN: 0022-2623

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

10578826

LANGUAGE: English  
OTHER SOURCE(S): CASREACT 147:469265  
GI

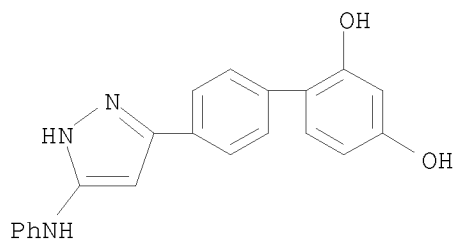


AB The cocrystal structure of a library hit was used to design a novel series of CHK1 inhibitors. The new series retained the critical hydrogen-bonding groups of the resorcinol moiety for binding but lacked the phenolic anilide moiety. The newly designed compds. I (X = CH, N; R = Me<sub>2</sub>CHNH, Me<sub>2</sub>N, pyrrolo, piperidino, cyclopropylamino, etc.) exhibited similar enzymic activity, while demonstrating increased cellular potency. I (X = CH, R = cyclopropylamino), showing no single agent effect, potentiated the antiproliferative effect of Gemcitabine in both prostate and breast cancer cell lines.

IT 838823-53-3P  
RL: PAC (Pharmacological activity); PRP (Properties); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)  
(cocrystal structure bound to CHK1 enzyme; structure-based design and preparation of (5-aryl-amino-3-pyrazolyl)biphenyls as human CHK1 inhibitors)

RN 838823-53-3 HCAPLUS

CN [1,1'-Biphenyl]-2,4-diol, 4'-[5-(phenylamino)-1H-pyrazol-3-yl]- (CA INDEX NAME)

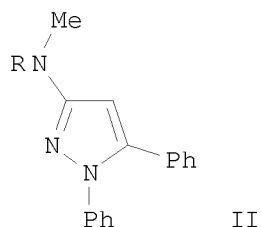
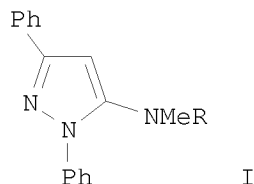


REFERENCE COUNT: 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 2 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN  
ACCESSION NUMBER: 2005:1122125 HCAPLUS  
DOCUMENT NUMBER: 144:36286  
TITLE: Highly Regioselective Synthesis of 1-Aryl-3 (or 5)-alkyl/aryl-5 (or 3)-(N-cycloamino)pyrazoles  
AUTHOR(S): Peruncheralathan, S.; Yadav, A. K.; Ila, H.; Junjappa, H.  
CORPORATE SOURCE: Department of Chemistry, Indian Institute of

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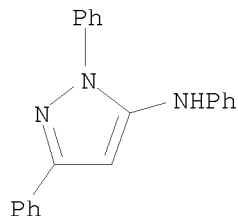
Technology, Kanpur, 208016, India  
SOURCE: Journal of Organic Chemistry (2005), 70(23), 9644-9647  
CODEN: JOCEAH; ISSN: 0022-3263  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 144:36286  
GI



AB An efficient highly regioselective protocol for the synthesis of isomeric 1,3-diaryl (or 1-aryl-3-alkyl) and 1,5-diaryl (or 1-aryl-5-alkyl)-5 (or 3)-(N-cycloamino)pyrazoles has been reported by cyclocondensation of common  $\alpha$ -oxoketene N,S-acetal precursors with arylhydrazines by variation of reaction conditions. E.g., reaction of  $\text{PhCOCH:C(SMe)NMeCH}_2\text{C}_6\text{H}_4\text{OMe-4}$  with  $\text{PhNHNH}_2$  in presence of NaH in DMF/ $\text{C}_6\text{H}_6$  gave 65% 5-aminopyrazole I ( $\text{R} = \text{CH}_2\text{C}_6\text{H}_4\text{OMe-4}$ ). On the other hand, reaction of  $\text{PhCOCH:C(SMe)NMeCH}_2\text{C}_6\text{H}_4\text{OMe-4}$  with  $\text{PhNHNH}_2$  in presence of DABCO gave 69% 3-aminopyrazole II (same R).

IT 94863-16-8P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(regioselective preparation of isomeric 1,3-diaryl (or 1-aryl-3-alkyl) and 1,5-diaryl (or 1-aryl-5-alkyl)-5 (or 3)-(N-cycloamino)pyrazoles by cyclocondensation of  $\alpha$ -oxoketene N,S-acetal precursors with arylhydrazines)

RN 94863-16-8 HCAPLUS  
CN 1H-Pyrazol-5-amine, N,1,3-triphenyl- (CA INDEX NAME)

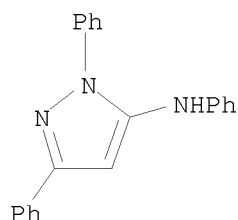


REFERENCE COUNT: 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

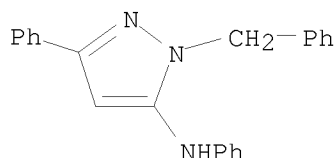
L4 ANSWER 3 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN  
ACCESSION NUMBER: 2005:419724 HCAPLUS

10578826

DOCUMENT NUMBER: 143:115479  
TITLE: Solid-Phase Synthesis of 5-Substituted Amino Pyrazoles  
AUTHOR(S): Dodd, Dharmpal S.; Martinez, Rogelio L.; Kamau, Muthoni; Ruan, Zheming; Van Kirk, Katy; Cooper, Christopher B.; Hermsmeier, Mark A.; Traeger, Sarah C.; Poss, Michael A.  
CORPORATE SOURCE: Early Discovery Chemistry New Leads Chemistry-Applied Biotechnology and Discovery Analytical Services, Bristol-Myers Squibb Pharmaceutical Research Institute, Princeton, NJ, 08543-4000, USA  
SOURCE: Journal of Combinatorial Chemistry (2005), 7(4), 584-588  
CODEN: JCCHFF; ISSN: 1520-4766  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 143:115479  
AB An efficient method for the solid-supported synthesis of 5-N-alkylamino and 5-N-arylamino pyrazoles is described. This method is general and mild and utilizes readily accessible resin-immobilized  $\beta$ -ketoamides as starting materials. Resin-immobilized  $\beta$ -ketoamide, aryl-, or alkylhydrazine and Lawesson's reagent are suspended in a mixture of THF/Py and heated at 50-55 °C to give a resin-bound 5-aminopyrazole, that is liberated from the solid support by treatment with TFA.  
IT 94863-16-8P 857636-66-9P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(solid-supported synthesis of 5-N-alkylamino and 5-N-arylamino pyrazoles using resin-immobilized  $\beta$ -ketoamides as starting materials)  
RN 94863-16-8 HCAPLUS  
CN 1H-Pyrazol-5-amine, N,1,3-triphenyl- (CA INDEX NAME)



RN 857636-66-9 HCAPLUS  
CN 1H-Pyrazol-5-amine, N,3-diphenyl-1-(phenylmethyl)- (CA INDEX NAME)



REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT



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L4 ANSWER 4 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2005:99354 HCAPLUS

DOCUMENT NUMBER: 142:198068

TITLE: Preparation of aminopyrazoles as CHK1 checkpoint protein kinase inhibitors.

INVENTOR(S): Johnson, Michael David; Teng, Min; Zhu, Jinjiang

PATENT ASSIGNEE(S): Pfizer Inc., USA

SOURCE: PCT Int. Appl., 127 pp.

CODEN: PIXXD2

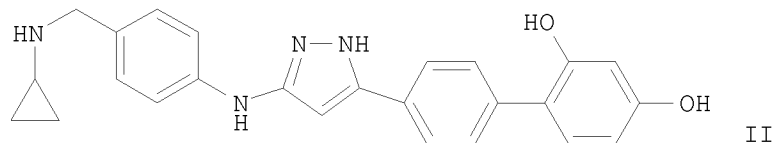
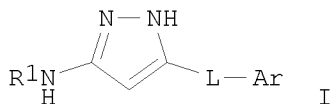
DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005009435	A1	20050203	WO 2004-IB2397	20040714
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
CA 2532231	A1	20050203	CA 2004-2532231	20040714
BR 2004012820	A	20060926	BR 2004-12820	20040714
JP 2006528661	T	20061221	JP 2006-521691	20040714
US 20050043381	A1	20050224	US 2004-897849	20040722
MX 2006000933	A	20060330	MX 2006-933	20060124
PRIORITY APPLN. INFO.:			US 2003-489976P	P 20030725
			WO 2004-IB2397	W 20040714
OTHER SOURCE(S):			CASREACT 142:198068; MARPAT 142:198068	
GI				

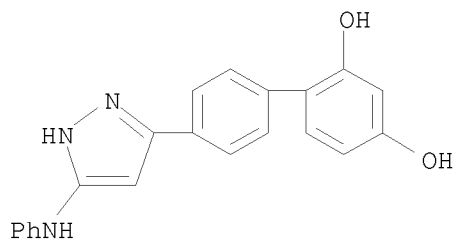


AB Title compds. [I; L = 5-6 membered (substituted) heterocyclylene; Ar = 5-6 membered (substituted) (hetero)aryl; R1 = (substituted) aryl(alkyl), heterocyclyl(alkyl), cycloalkyl(alkyl), alkenyl, alkyl; R2 = H, halo, (substituted) alkyl], were prepared Thus, title compound (II) (preparation outlined) inhibited human CHK1 with Ki <1 nM.

IT 838823-53-3P  
 RL: PAC (Pharmacological activity); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)  
 (claimed compound; preparation of aminopyrazoles as CHK1 checkpoint protein kinase inhibitors)

RN 838823-53-3 HCAPLUS

CN [1,1'-Biphenyl]-2,4-diol, 4'-[5-(phenylamino)-1H-pyrazol-3-yl]- (CA INDEX NAME)



REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 5 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2004:385028 HCAPLUS

DOCUMENT NUMBER: 141:123593

TITLE: One-pot synthesis of 5-(substituted-amino)pyrazoles

AUTHOR(S): Dodd, Dharmpal S.; Martinez, Rogelio L.

CORPORATE SOURCE: Squibb Pharmaceutical Research Institute, Early Discovery Chemistry, Bristol-Myers, Princeton, NJ, 08543-4000, USA

SOURCE: Tetrahedron Letters (2004), 45(22), 4265-4267  
 CODEN: TELEAY; ISSN: 0040-4039

PUBLISHER: Elsevier

DOCUMENT TYPE: Journal

LANGUAGE: English

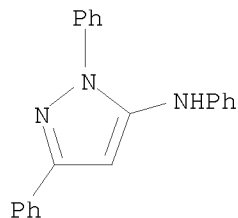
OTHER SOURCE(S): CASREACT 141:123593

AB An efficient and mild one-pot synthesis of substituted 5-alkylamino and/or 5-(arylamino)pyrazoles is described. A suitably decorated  $\beta$ -keto amide, an aryl or alkyl hydrazine and Lawesson's reagent are suspended in THF/Py and gently heated to yield the requisite 5-aminopyrazoles. For example, the reaction of N,N-diethyl-3-oxobutanamide with (phenyl)hydrazine in the presence of Lawesson's reagent gave N,N-diethyl-3-methyl-1-phenyl-1H-pyrazol-5-amine in 95% yield. It is postulated that this method should also be easily adaptable for automated parallel synthesis.

IT 94863-16-8P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (one-pot synthesis of pyrazolamines from  $\beta$ -oxo amides and hydrazines in presence of Lawesson's reagent)

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RN 94863-16-8 HCAPLUS  
CN 1H-Pyrazol-5-amine, N,1,3-triphenyl- (CA INDEX NAME)



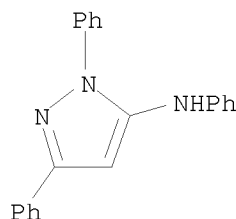
REFERENCE COUNT: 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 6 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN  
ACCESSION NUMBER: 2002:917721 HCAPLUS  
DOCUMENT NUMBER: 138:146744  
TITLE: 1,3-Diphenyl-1H-pyrazolo[3,4-b]quinoline: A Versatile Fluorophore for the Design of Brightly Emissive Molecular Sensors  
AUTHOR(S): Rurack, Knut; Danel, Andrzej; Rotkiewicz, Krystyna; Grabka, Danuta; Spieles, Monika; Rettig, Wolfgang  
CORPORATE SOURCE: Department I.3902, Federal Institute for Materials Research and Testing (BAM), Berlin, D-12489, Germany  
SOURCE: Organic Letters (2002), 4(26), 4647-4650  
CODEN: ORLEF7; ISSN: 1523-7060  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The 1,3-diphenyl-1H-pyrazolo[3,4-b]-quinoline chromophore is a versatile building block for the construction of brightly fluorescent mol. sensors. Facile synthetic procedures allow integration of the chromophore into fluorophore-spacer-receptor systems as well as fluoroionophores operating via intramol. charge transfer. Whereas the former photoinduced electron-transfer probes show strong analyte-induced fluorescence enhancement, the latter exhibit bright ratiometric dual emission. Employing prototype macrocyclic receptors, the favorable signaling features for metal ion recognition are demonstrated.

IT 94863-16-8  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(1,3-di-Ph-1H-pyrazolo[3,4-b]quinoline as versatile fluorophore for design of brightly emissive mol. sensors)

RN 94863-16-8 HCAPLUS  
CN 1H-Pyrazol-5-amine, N,1,3-triphenyl- (CA INDEX NAME)



REFERENCE COUNT: 56 THERE ARE 56 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 7 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2001:545674 HCAPLUS

DOCUMENT NUMBER: 135:137516

TITLE: Synthesis of heteroarylbenzamides and analogs used for inhibiting protein kinases

INVENTOR(S): Bender, Steven Lee; Bhumralkar, Dilip; Collins, Michael Raymond; Cripps, Stephan James; Deal, Judith Gail; Nambu, Mitchell David; Palmer, Cynthia Louise; Peng, Zhengwei; Varney, Michael David; Jia, Lei

PATENT ASSIGNEE(S): Agouron Pharmaceuticals, Inc., USA

SOURCE: PCT Int. Appl., 237 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001053274	A1	20010726	WO 2001-US1723	20010119
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
CA 2394703	A1	20010726	CA 2001-2394703	20010119
US 20020103203	A1	20020801	US 2001-764306	20010119
US 6635641	B2	20031021		
EP 1252146	A1	20021030	EP 2001-906592	20010119
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
BR 2001008025	A	20021105	BR 2001-8025	20010119
JP 2003529558	T	20031007	JP 2001-553276	20010119
MX 2002007102	A	20030128	MX 2002-7102	20020719
US 20040092747	A1	20040513	US 2003-621979	20030717
PRIORITY APPLN. INFO.:			US 2000-177059P	P 20000121
			US 2001-764306	A3 20010119
			WO 2001-US1723	W 20010119

OTHER SOURCE(S): MARPAT 135:137516

GI

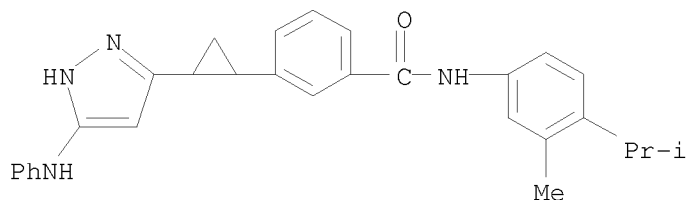
\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

AB Title compds. I [Z = CH, NH; Q = moiety such that ring A is (un)substituted mono- or bicyclic heteroaryl which has at least 2 carbon atoms in the heteroaryl ring system; X = CH<sub>2</sub>, O, S, NH; Y = CH<sub>2</sub>, O, S, provided at least one of X and Y = CH<sub>2</sub> or X and Y form a cyclopropyl ring; R<sub>2</sub>-3 = H, Me, halo, CF<sub>3</sub>, CN; R<sub>4</sub> = CONHR<sub>5</sub>, NHCOR<sub>6</sub>; where R<sub>5</sub> = (un)substituted aryl, heteroaryl, cycloalkyl, etc.; R<sub>6</sub> = (un)substituted aryl, heteroaryl, cycloalkyl, etc] are prepared Examples include synthetic procedures for over 150 compds., 11 biol. assays and 3 sample formulations. For instance, 3-mercaptobenzoic acid was treated with  $\alpha$ -chloro-N-methoxy-N-methylacetamide followed by carbodiimide coupling to 2-methyl-6-aminoquinoline to give II. II was converted to a  $\beta$ -thiono-ketone with thioacetanilide/n-BuLi followed by treatment with hydrazine to give pyrazole III. III gave 85% inhibition of an lck protein tyrosine kinase at 5  $\mu$ M and had K<sub>i</sub> = 2.21 nM for VEGF-R2A50. Treatment of cancer as well as other disease states associated with unwanted angiogenesis and/or cellular proliferation, such as diabetic retinopathy, neovascular glaucoma, rheumatoid arthritis, and psoriasis are claimed uses of the invention.

IT 351320-34-8P  
 RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)  
 (synthesis of heteroarylbenzamides used for inhibiting protein kinases)

RN 351320-34-8 HCAPLUS

CN Benzamide, N-[3-methyl-4-(1-methylethyl)phenyl]-3-[2-[5-(phenylamino)-1H-pyrazol-3-yl]cyclopropyl]- (CA INDEX NAME)



REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

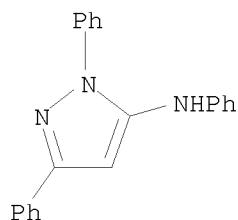
L4 ANSWER 8 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN  
 ACCESSION NUMBER: 2000:673725 HCAPLUS  
 DOCUMENT NUMBER: 134:71524  
 TITLE: Microwave-assisted, facile route to 1H-pyrazolo[3,4-b]quinolines  
 AUTHOR(S): Danel, Andrzej; Chaczatrian, Karen; Tomasik, Piotr  
 CORPORATE SOURCE: Dep. of Chem., Univ. of Agriculture, Krakow, 31 120, Pol.  
 SOURCE: ARKIVOC [online computer file] (2000), 1(1), 51-57  
 CODEN: AKVCFI

URL: [http://www.arkat-usa.org/ARKIVOC/JOURNAL\\_CONTENT/manuscripts/2000/00-2107CP%20as%20published%20mainmanuscript.pdf](http://www.arkat-usa.org/ARKIVOC/JOURNAL_CONTENT/manuscripts/2000/00-2107CP%20as%20published%20mainmanuscript.pdf)

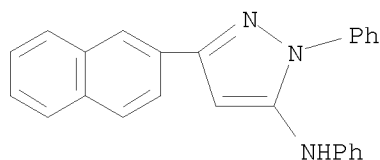
PUBLISHER: ARKAT Foundation  
 DOCUMENT TYPE: Journal; (online computer file)  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 134:71524

AB Aromatic aldehydes have been reported to react with 5-anilinopyrazoles in the presence of ZnCl<sub>2</sub> to give the corresponding benzylidenopyrazoles. In this paper evidence is given that the corresponding products are, in fact, 1H-pyrazolo[3,4-b]quinolines. This observation opens a novel route to these compds. They show a blue emission in the solid state and, therefore, they are useful blue luminophores for electroluminescent devices. The synthetic procedure reported in the literature was significantly modified and improved by application of microwave heating. In our modified synthesis the reaction time was reduced from the usual 5 to 8 h to 5 to 7 min and the reaction products were formed without contamination.

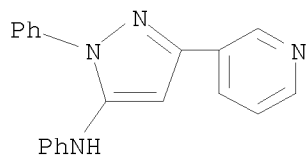
IT 94863-16-8P 314274-99-2P 314275-01-9P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 94863-16-8 HCAPLUS  
 CN 1H-Pyrazol-5-amine, N,1,3-triphenyl- (CA INDEX NAME)



RN 314274-99-2 HCAPLUS  
 CN 1H-Pyrazol-5-amine, 3-(2-naphthalenyl)-N,1-diphenyl- (CA INDEX NAME)



RN 314275-01-9 HCAPLUS  
 CN 1H-Pyrazol-5-amine, N,1-diphenyl-3-(3-pyridinyl)- (CA INDEX NAME)



REFERENCE COUNT: 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 9 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1996:1287 HCAPLUS

DOCUMENT NUMBER: 124:202094

ORIGINAL REFERENCE NO.: 124:37361a,37364a

TITLE: Synthesis and biological activity of some new pyrazolyl-1,8-naphthyridines

AUTHOR(S): Rani, H. Shailaja; Mogilaiah, K.; Sreenivasulu, B.

CORPORATE SOURCE: Department Chemistry, Kakatiya University, Warangal, 506 009, India

SOURCE: Indian Journal of Heterocyclic Chemistry (1995), 5(1), 45-8

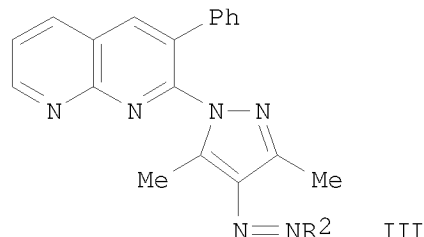
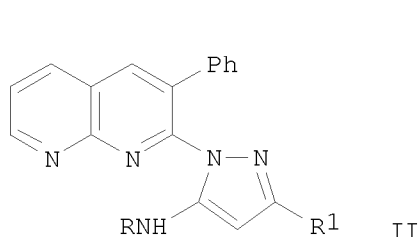
CODEN: IJCHEI; ISSN: 0971-1627

PUBLISHER: Lucknow University, Dep. of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

GI



AB 2-Hydrazino-3-phenyl-1,8-naphthyridine (I) when heated with acetylacetone and Et acetoacetate gave 2-(3,5-dimethylpyrazol-1-yl)-3-phenyl-1,8-naphthyridine and 3-methyl-1-(3-phenyl-1,8-naphthyridin-2-yl)-5(4H)-pyrazolone. I was treated with acetoacetanilides/benzoylacetanilides and cyclized to give 2-(5-arylamino-3-methyl/phenylpyrazol-1-yl)-3-phenyl-1,8-naphthyridines II (R = Ph, substituted phenyl; R1 = Me, Ph). Cyclocondensation of I with arylazoacetylacetones gave the 2-(4-arylazo-3,5-dimethylpyrazol-1-yl)-3-phenyl-1,8-naphthyridines III (R2 = Ph, substituted phenyl). The compds. have been characterized on the basis of their elemental analyses and spectral data and tested for their antibacterial and antifungal activities.

IT 174137-80-5P

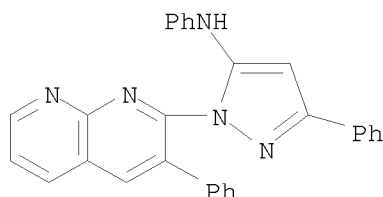
RL: BAC (Biological activity or effector, except adverse); BSU (Biological

study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)

(preparation and antimicrobial activity of some new 2-(pyrazol-1-yl)-1,8-naphthyridines)

RN 174137-80-5 HCAPLUS

CN 1H-Pyrazol-5-amine, N,3-diphenyl-1-(3-phenyl-1,8-naphthyridin-2-yl)- (CA INDEX NAME)



L4 ANSWER 10 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1994:217592 HCAPLUS

DOCUMENT NUMBER: 120:217592

ORIGINAL REFERENCE NO.: 120:38641a,38644a

TITLE: Synthesis and reactivity of 6H-1,3,4-selenadiazines

AUTHOR(S): Pfeiffer, W. D.; Rossberg, H.

CORPORATE SOURCE: Fachrichtung Chem., Ernst-Mortiz-Arndt-Univ., Greifswald, Germany

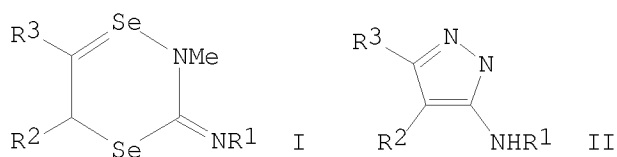
SOURCE: Pharmazie (1993), 48(10), 732-5

CODEN: PHARAT; ISSN: 0031-7144

DOCUMENT TYPE: Journal

LANGUAGE: German

GI



AB The 6H-1,3,4-selenadiazines I [R1 =Pr, CHMe2, CMe3, Ph; R2 = H, Me, Ph,; R3 = Me, Ph, 4-BrC6H4, 4-ClC6H4, 4-MeC6H4, 4-FC6H4] were prepared by condensation of  $\alpha$ -halo ketones and H2NNMeCSeNHR1. I were converted to pyrazoles II by selenium elimination in boiling glacial acetic acid. Kinetic measurements show that I are much slower to undergo ring contraction than thiadiazines.

IT 153849-11-7P

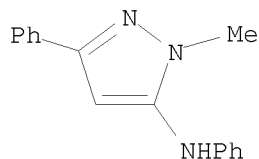
RL: FORM (Formation, nonpreparative); PREP (Preparation)

(formation of, by ring contraction of selenadiazine)

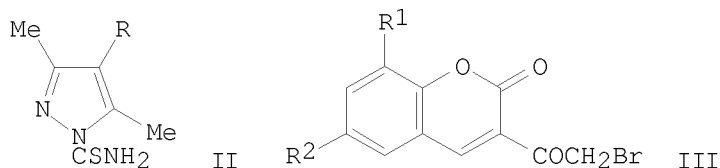
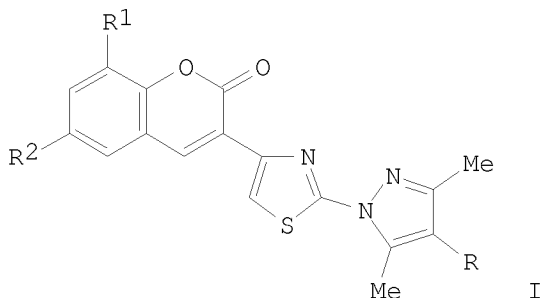
RN 153849-11-7 HCAPLUS

CN 1H-Pyrazol-5-amine, 1-methyl-N,3-diphenyl- (CA INDEX NAME)





L4 ANSWER 11 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN  
 ACCESSION NUMBER: 1988:528892 HCAPLUS  
 DOCUMENT NUMBER: 109:128892  
 ORIGINAL REFERENCE NO.: 109:21473a,21476a  
 TITLE: Studies on coumarin derivatives. Part V. Synthesis of a new type of pyrazolothiazole  
 AUTHOR(S): Ravinder, P.; Rao, V. Rajeswar; Rao, T. V. Padmanabha  
 CORPORATE SOURCE: Dep. Chem., Kakatiya Univ., Warangal, 506 009, India  
 SOURCE: Collection of Czechoslovak Chemical Communications (1988), 53(2), 336-9  
 CODEN: CCCCAK; ISSN: 0366-547X  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 109:128892  
 GI



AB Eighteen of the title pyrazolothiazoles, e.g. I (R = H, PhN:N, 4-MeC6H4N:N, R1 = H, R2 = H, Br; same R, R1 = R2 = Br), were prepared in 70-80% yield by cyclocondensation of thiocarbamoylpyrazoles II with coumarins III.

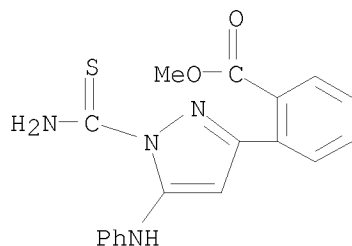
IT 116317-18-1

RL: RCT (Reactant); RACT (Reactant or reagent)  
 (cyclocondensation reaction of, with (bromoacetyl)coumarins, pyrazolothiazole derivs. from)

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RN 116317-18-1 HCAPLUS

CN Benzoic acid, 2-[1-(aminothioxomethyl)-5-(phenylamino)-1H-pyrazol-3-yl]-, methyl ester (CA INDEX NAME)

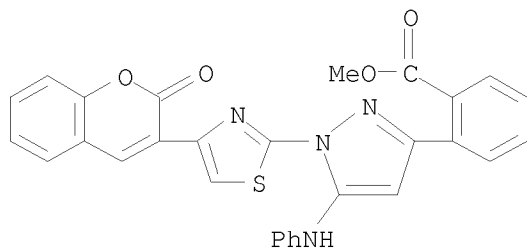


IT 116317-13-6P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (preparation and acetylation of)

RN 116317-13-6 HCAPLUS

CN Benzoic acid, 2-[1-[4-(2-oxo-2H-1-benzopyran-3-yl)-2-thiazolyl]-5-(phenylamino)-1H-pyrazol-3-yl]-, methyl ester (CA INDEX NAME)



L4 ANSWER 12 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1972:475162 HCAPLUS

DOCUMENT NUMBER: 77:75162

ORIGINAL REFERENCE NO.: 77:12419a,12422a

TITLE: Propiolamidines. I. Synthesis of N,N'-disubstituted phenylpropiolamidines and new routes to 5-N-substituted amino-3-phenylisoxazoles and 5-N-substituted amino-1,3-diphenylpyrazoles  
AUTHOR(S): Fujita, Hiroshi; Endo, Rokuro; Aoyama, Akira; Ichii, Takeshi

CORPORATE SOURCE: Cent. Res. Lab., Sankyo Co., Ltd., Tokyo, Japan  
SOURCE: Bulletin of the Chemical Society of Japan (1972), 45(6), 1846-52

CODEN: BCSJA8; ISSN: 0009-2673

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 77:75162

AB N, N'-Disubstituted phenylpropiolamidines were synthesized from phenylacetylene and carbodiimides. They were inert toward nucleophiles in a neutral or basic medium, but reactive in an acidic one. They reacted in

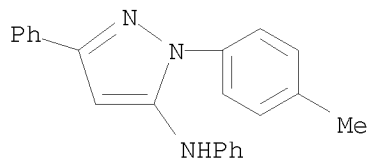
the presence of HCl with HONH<sub>2</sub>, NH<sub>2</sub>NH<sub>2</sub>, and arylhydrazines to give 5-N-substituted amino-3-phenylisoxazoles, 5-N-substituted amino-3-phenylpyrazole and 5-N-substituted amino-1-aryl-3-phenylpyrazoles, resp., by nucleophilic addition followed by cyclization.

IT 36988-04-2P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 36988-04-2 HCAPLUS

CN 1H-Pyrazol-5-amine, 1-(4-methylphenyl)-N,3-diphenyl- (CA INDEX NAME)



L4 ANSWER 13 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1963:415565 HCAPLUS

DOCUMENT NUMBER: 59:15565

ORIGINAL REFERENCE NO.: 59:2795c-h,2796a-c

TITLE: Study of the  $\beta$ -oxo thioanilides. I. Reactions  
with arylhydrazines

AUTHOR(S): Pocar, Donato; Bianchetti, Giuseppe; Maiorana, Stefano  
CORPORATE SOURCE: Univ. Milan

SOURCE: Gazzetta Chimica Italiana (1963), 93, 100-13  
CODEN: GCITA9; ISSN: 0016-5603

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB The reactions of PhNHNH<sub>2</sub> (I), p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHNH<sub>2</sub> (II), oO<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHNH<sub>2</sub> (III), and 2,4-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NHNH<sub>2</sub> (IV) with several  $\beta$ -oxo thioacid anilides yielded in all cases the corresponding arylhydrazones which frequently can be isolated in substance and then cyclized by various methods to pyrazoles. The tendency of the arylhydrazones to cyclize is correlated with the structural characteristics, such as steric hindrance and strain. It is demonstrated that the compound synthesized by Worall (CA 14, 1832) and by Huenig, et al. (CA 57, 4653e), from I and BzSCH<sub>2</sub>CO<sub>2</sub>NHPh (V) is 1,3-diphenyl-5-phenylaminopyrazole (VI). V (2.55 g.) in 15 cc. 80% AcOH refluxed 4 hrs. with 1.08 g. I in 50% AcOH and evaporated gave VI, m. 153° (MeOH). 1,3-Diphenyl-2-methyl-5-chloropyrazole-HI (7.93 g.), 3.72 g. PhNH<sub>2</sub> heated 4 hrs. at 200° in a sealed tube also yielded VI. AcCH<sub>2</sub>-CSNHPh (VII) (1.93 g.) in 20 cc. 70% AcOH treated with 1.08 g. PhNHNH<sub>2</sub>, refluxed 2 hrs., cooled, and evaporated yielded the 3-Me analog of VI, m. 119-20°. 1-Cyclohexane-2-thiocarboxylic anilide (VIII) (2.33 g.) and 1.12 g. I mixed without solvent and diluted after a few min. with ligroine, and the oil layer washed with H<sub>2</sub>O, dissolved with warming in EtOH, and cooled gave the phenylhydrazone (IX) of VIII, m. 138° (decomposition). IX refluxed 1 hr. in 90% AcOH (H<sub>2</sub>S is evolved) yielded 2-phenyl-3-phenylamino-4,5,6,7-tetrahydroindazole (X), m. 158° (MeOH), also obtained by refluxing equimolar amts. of VIII and I during 2 hrs. in 60% AcOH. 1-Cyclopentanone-2-thiocarboxylic anilide (XI) (2.19 g.) in 10 cc. EtOH and 1.08 g. I kept at room temperature overnight yielded the phenylhydrazone (XII) of XI, m. 150-1° (decomposition) (EtOH). XII

refluxed in AcOH gave 2-phenyl-3-phenylamino-4,5-dihydrocyclopenta[c]pyrazole (XIII), m. 148°, also obtained by heated equimolar amts. of I and XI in 60% AcOH during 3 hrs. V (5.1 g.) in 20 cc. EtOH treated with 3.06 g. II in 60 cc. hot 50% AcOH, refluxed 1 min., and filtered yielded the 1-(p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>) analog of VI, yellow crystals, m. 203°; the filtrate cooled yielded the red, crystalline 4-nitrophenylhydrazone of V, m. 200°, changing to yellow at 150-3°. VII (1.93 g.) in 10 cc. EtOH refluxed 0.5 hr. with 1.53 g. II in 50% AcOH and evaporated, the residue heated with 20% HCl, treated with C, and cooled, and the resulting 1-(p-nitrophenyl)-3-methyl-5-phenylaminopyrazole-HCl (XIV.HCl), pale yellow crystals, m. 188-93°, suspended in H<sub>2</sub>O, treated with aqueous K<sub>2</sub>CO<sub>3</sub>, and extracted with Et<sub>2</sub>O yielded XIV, light yellow, m. 111-12° (ligroine). VIII (2.33 g.) and 1.53 g. II in 50 cc. 50% AcOH heated 0.5 hr. yielded the 2-(p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>) analog of X, golden-yellow flakes, m. 135-6° (ligroine). XI (2.19 g.) and 1.53 g. II in 50 cc. 50% AcOH and 40 cc. EtOH refluxed 5 min. gave the p-nitrophenylhydrazone (XV) of XI, dark yellow needles, m. 160-1° (EtOH). XV and 1 equivalent Pb(OAc)<sub>2</sub>·3H<sub>2</sub>O in 50% AcOH refluxed 1 hr., filtered hot, and evaporated, and the residue washed with H<sub>2</sub>O, dissolved in Et<sub>2</sub>O, and evaporated gave the golden-yellow 1-(p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>) analog of XIII, m. 146-7° (ligroine). V (2.55 g.) in 10 cc. EtOH refluxed, treated with 1.53 g. III in 30 cc. 50% AcOH, refluxed 5 min., and worked up yielded the o-nitrophenylhydrazone (XVI) of V, m. 179° (decomposition) (EtOAc-petr. ether). XVI (3.90 g.) in 40 cc. AcOH treated with 3.80 g. Pb(OAc)<sub>2</sub>·3H<sub>2</sub>O in 15 cc. 50% AcOH, refluxed 0.5 hr., filtered, and diluted with an equal volume H<sub>2</sub>O yielded the yellow 1-(o-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>) analog of VI, m. 164-5° (EtOH). VII (1.93 g.) in 10 cc. hot EtOH treated with 1.53 g. III in 25 cc. 50% AcOH and refluxed 5 min. yielded the orange o-nitrophenylhydrazone (XVII) of VII, m. 129-30° (EtOAc-petr. ether). XVII (3.28 g.) in 50 cc. AcOH refluxed 10 min. with 3.80 g. Pb(OAc)<sub>2</sub>·3H<sub>2</sub>O in 20 cc. 50% AcOH gave the yellow 1-(o-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>) analog of VI, m. 130° (ligroine). VIII (2.33 g.) in 10 cc. EtOH refluxed 1 min. with 1.53 g. III in 30 cc. 50% AcOH yielded the yellow-orange o-nitrophenylhydrazone (XVIII) of VIII, m. 171-2° (EtOAc-petr. ether). XVIII (3.68 g.) in 100 cc. AcOH refluxed 20 min. with 3.80 g. Pb(OAc)<sub>2</sub>·3H<sub>2</sub>O in 20 cc. 50% AcOH gave the o-isomer of XIV, golden-yellow flakes, m. 165-6° (EtOH). XI (2.19 g.) in 20 cc. EtOH and 1.53 g. III in 40 cc. 50% AcOH refluxed 5 min. yielded the o-nitrophenylhydrazone of XI, red crystals, m. 118° (EtOH). V (2.55 g.) in 15 cc. EtOH and 1.98 g. IV in 50 cc. 70% AcOH refluxed 5 min. yielded the 2,4-dinitrophenylhydrazone (XIX) of V, m. 184° (decomposition) (EtOAc-petr. ether). XIX (2.17 g.) in 30 cc. AcOH refluxed 15 min. with 1.99 g. Pb(OAc)<sub>2</sub>·3H<sub>2</sub>O in 15 cc. 50% AcOH gave the 1-[2,4-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>] analog of VI, yellow-brown crystals, m. 216-17° (EtOH). VII (1.93 g.), 1.98 g. IV, and 30 cc. 60% AcOH, refluxed, diluted with EtOH to turbidity, refluxed 1 min., cooled, and filtered yielded the 2,4-dinitrophenylhydrazone (XX) of VII, golden-yellow flakes, m. 178-9° (EtOAc-Et<sub>2</sub>O). XX (3.73 g.) in 50 cc. refluxing AcOH treated with 3.80 g. Pb(OAc)<sub>2</sub>·3H<sub>2</sub>O in 15 cc. refluxing 50% AcOH, refluxed 15 min., filtered, and diluted with H<sub>2</sub>O, and the tacky precipitate dissolved in CHCl<sub>3</sub> and

repptd. with petr. ether gave the 1-[2,4-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>] analog of XIV, dark orange needles, m. 156° (ligroine). VIII (2.33 g.) in 10 cc. EtOH refluxed with 1.89 g. IV in 40 cc. 70% AcOH, diluted with a few cc. EtOH to turbidity, and cooled after 20 min. yielded the golden-yellow 2,4-dinitrophenylhydrazone of VIII, m. 167° (EtOAc-petr. ether). XI

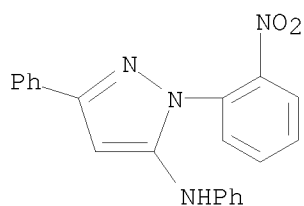
(2.19 g.) with 1.98 g. IV refluxed 10 min. in 15 cc. EtOH and 40 cc. 70% AcOH yielded the 2,4-dinitrophenylhydrazone of XI, yellow-orange crystals, m. 167° (EtOH).

IT 88844-15-9P, Pyrazole, 5-anilino-1-(o-nitrophenyl)-3-phenyl-  
 88844-16-0P, Pyrazole, 5-anilino-1-(p-nitrophenyl)-3-phenyl-  
 94863-16-8P, Pyrazole, 5-anilino-1,3-diphenyl- 94878-85-0P  
 , Pyrazole, 5-anilino-1-(2,4-dinitrophenyl)-3-phenyl-  
 RL: PREP (Preparation)

(preparation of)

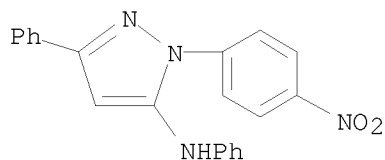
RN 88844-15-9 HCAPLUS

CN 1H-Pyrazol-5-amine, 1-(2-nitrophenyl)-N,3-diphenyl- (CA INDEX NAME)



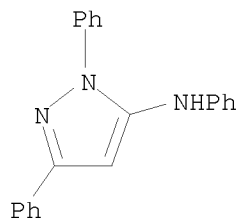
RN 88844-16-0 HCAPLUS

CN 1H-Pyrazol-5-amine, 1-(4-nitrophenyl)-N,3-diphenyl- (CA INDEX NAME)



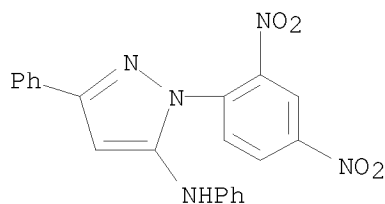
RN 94863-16-8 HCAPLUS

CN 1H-Pyrazol-5-amine, N,1,3-triphenyl- (CA INDEX NAME)



RN 94878-85-0 HCAPLUS

CN 1H-Pyrazol-5-amine, 1-(2,4-dinitrophenyl)-N,3-diphenyl- (CA INDEX NAME)



L4 ANSWER 14 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1963:33419 HCAPLUS

DOCUMENT NUMBER: 58:33419

ORIGINAL REFERENCE NO.: 58:5692b-g

TITLE: Ketene derivatives. V. Oxalylketene mercaptals and related compounds

AUTHOR(S): Stachel, Hans Dietrich

CORPORATE SOURCE: Univ. Marburg, Germany

SOURCE: Chemische Berichte (1962), 95, 2166-71

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 58:33419

GI For diagram(s), see printed CA Issue.

AB cf. CA 58, 4540b. [(EtO)2C:CHCO]2 (I) was converted with suitable mercaptans to oxalylketene O,S-acetals or oxalylketene mercaptals which were also prepared from CH2:C(OEt)SEt (II) or CH2:C(SEt). (III), resp., with (COCl)2 (IV). I (1.4 g.) and 3 cc. PhCH2SH heated slowly to about 170°, kept several min. at 175°, cooled, diluted with 4 vols. EtOH, and filtered after 0.5 hr. gave 1.1 g. yellow [OCCH:C(OEt)SCH2Ph]2 (V), decomposed about 190°. V and piperidine refluxed 2 min. gave yellow oxalylketene tetrapiperidinoaminal. II (3.1 g.) in 15 cc. dry Et2O treated at 0° with 0.5 cc. IV, kept 15 min. at room temperature, and filtered gave 0.9 g. yellow [OCCH:C(OEt)SEt]2 (VI), m. 154-5°. VI (0.2 g.) shaken with EtOH and kept 5 days at room temperature with an equal weight

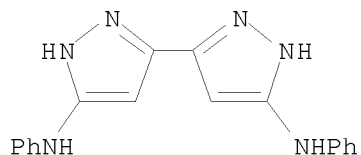
PhNH2 gave yellow oxalylketene dianilino-O,N-acetal (VII), m. 160-2° (Ac2O). VI (0.5 g.) in EtOH and 5 drops concentrated HCl kept 3 days and evaporated at room temperature gave (COCH2CO2Et)2, m. 78-80°. I (1.4 g.) and 2 cc. (CH2SH)2 warmed to beginning reaction, diluted after 2-3 min. with 2 vols. EtOH, and filtered after 0.5 hr. yielded 0.3 g. yellow oxalylketene bis(ethylene)mercaptal, m. 260° (decomposition) (2:1 AcOH-HCONMe2). (EtS)2C:CHCOCOC1 (VIII) (2.4 g.) in 120 cc. dry Et2O treated with 3.7 g. III and kept 24 hrs. at room temperature yielded 0.9-1.0 g. [OCCH:C(SEt)2]2 (IX), m. 160-1° (Ac2O). VIII (2.4 g.) in about 10 cc. dry Et2O and 3.0 g. III kept overnight and filtered gave 75 mg. IX; the filtrate cooled gave 1.3 g. EtSCOCOCH:C(SEt)2 (X). VIII (2.4 g.) added to 3.7 g. III and 1.27 g. iodine in 20 cc. dry Et2O, kept 24 hrs. at room temperature, filtered, and the residue treated dropwise with piperidine left 0.65 g. IX undissolved; the mother liquor cooled gave 1.1 g. mixture of VIII and X. II (1.5 g.) in 5 cc. dry Et2O treated at -50° with 0.5 cc. IV and filtered, the residue added to excess CH2N2-Et2O, the mixture evaporated, and the crude product dissolved in a few cc. Et2O, filtered from the insol. polymethylene, and cooled to -50° gave 150 mg. yellow EtS(EtO)C:CHCOCOCHN2, m. 100-1°. VIII (0.5 g.) warmed briefly in H2O-containing dioxane and evaporated yielded 0.35 g. yellow (EtS)2C:CHCOCO2H,

m.

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about 125° (decomposition) (iso-Pr<sub>2</sub>O), which with CH<sub>2</sub>N<sub>2</sub> gave the Me ester. IX (0.5 g.) in about 10 cc. boiling PrOH treated dropwise with 0.5 g. N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O, refluxed about 15 min., and evaporated gave 250 mg. brownish yellow XI, m. 155-6° (MeOH). VII (0.5 g.) in PrOH treated dropwise with 10 drops N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O, heated about 3 min., filtered, and cooled gave 200 mg. red 3,3'-bis(5-anilinopyrazole), m. 265-8° (1:1 HCONMe<sub>2</sub>-H<sub>2</sub>O). III in EtOH with ale. iodine yielded black, powdery III.I<sub>2</sub>, decomposed 85-90°.

IT 98494-86-1P, 3,3'(or 5,5')-Bipyrazole, 5,5'(or 3,3')-dianilino-  
RL: PREP (Preparation)  
(preparation of)  
RN 98494-86-1 HCAPLUS  
CN [3,3'-Bi-1H-pyrazole]-5,5'-diamine, N5,N5'-diphenyl- (CA INDEX NAME)



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COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
93.21	279.31

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE	TOTAL
ENTRY	SESSION
-11.48	-11.48

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